PHYSICO-CHEMICAL CHANGES IN VALENCIA ORANGE CONCENTRATE DURING COMMERCIAL SCALE VACUUM CONCENTRATION

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ABSTRACT

Changes in refractometric solids (°Brix), viscosity, spectral reflectance (colour), *pH*, acidity, °Brix/acid ratio, colour, true ascorbic acid and carotene during commercial scale vacuum concentration of Valencia orange juice in a forced circulation, single-pass, falling-film Evaporator have been reported. With the advancing concentration, there was a gradual increase in °Brix, acidity, viscosity, colour, ascorbic acid and carotene, no change in °Brix/acid ratio but a slight fall in *pH*. Upto 4-fold concentration of the juice, the increase in viscosity was comparatively slight, but after 5-fold concentration, there was a steep rise in viscosity. There were negligible losses in carotene while the losses in true ascorbic acid were well within 5% during concentration up to 63° Brix.

Use of mid-season, healthy ripe oranges for juice extraction and concentration of the flash-heated juice (195°—200° F for 10—12 seconds) upto 5-fold concentration are suggested.

Introduction

Hitherto, little systematic attempt appears to have been made to study the effect of commercial scale vacuum concentration on the important physico-chemical aspects of Orange Juice at different stages of concentration. Curl [1] did report partial analysis of orange juice (concentrated at 110°F and pressure 40 mm) at 7 different stages of concentration but the report did not cover some of the most important aspects of concentration e.g., viscosity, *pH* etc. Besides, he reported changes in total ascorbic acid (and not true ascorbic acid). Recently, Siddappa and Bhatia[2] reported only ascorbic acid changes during laboratory scale vacuum concentration (in a small glass apparatus at 50—52°C, 40 mm. pressure) of 3 varieties of Indian Oranges (one of tight-skinned orange and 2 of mandarins). Evidently, the conditions of concentration described by them are much different from those in actual commercial practice. The present report covers practically all important aspects, *viz.*, changes in Refractometric solids, viscosity, reflectance (colour), pH, acidity, true ascorbic acid and carotene during commercial scale vacuum concentration of Valencia Orange Juice—most commonly employed for the purpose all over the world. The studies were made on several occasions in the 1956-57 season in one of the modern citrus concentrate factory in Gosford, N.S. Wales, Australia, handling 8—10 tons of oranges every day and producing 100 gallons of 4-fold concentrate per day, in addition to several hundred cases of orange squash and cordial. Only results from a typical run are presented in this paper.

Experimental Procedure

Raw Material—Valencia Oranges, being comparatively free from bitterness and being rich in flavour, were employed. Small to medium size, fairly uniformly ripe fruit, purchased in bulk lots of 8—10 tons per day direct from the fruit growers, were unloaded promptly from the delivery trucks, transferred to storage bins @ 50 boxes per bin, and conveyed past the trained inspectors who removed the undesirable fruit.

Juice Extraction—The selected healthy ripe fruits were fed to a battery of 6 rumblers. While the fruits were being washed with heavy sprays of water, they were rumbled for 2-3 minutes, depending upon the condition of the fruits. The water-oil emulsion was conveyed to the storage tank for subsequent separation and recovery of orange oil as a bye-product.

The cleanly rasped fruits were then automatically fed to a battery of 6 Smith and Searls juice extractors where the fruits were automatically quartered, internal pulp separated from peel and the pulp was squeezed in the screw-type extractor. The wet waste comprising peel and rag was conveyed to the waste line and later fed to dairy cattle. The coarse pulp from the juice was removed by passing the juice over vibrating screens (1/8″ mesh). About 10 per cent. of this coarsely screened juice was reserved separately in a stainless steel tank and the rest was further screened through another vibrating screen (0·027″ mesh) and stored in stainless steel tanks, till delivered to the evaporator through the flash-pasteurizer.

Vacuum Concentration—The juice was flash heated in an A.P.V. flash-pasteurizer to 195—200°F for 10—12 seconds with a view to inactivate the pectin methyl esterase as well as most of the contaminating micro-organisms, and then concentrated in a turbulent film single-pass vacuum evaporator (plate) described in detail elsewhere[3]. A positive centrifugal pump was placed just below the evaporator, with the aid of which the concentrate could be removed from the evaporator without breaking the vacuum and fresh juice from the flash-pasteurizer could be drawn in under vacuum so that the operation was

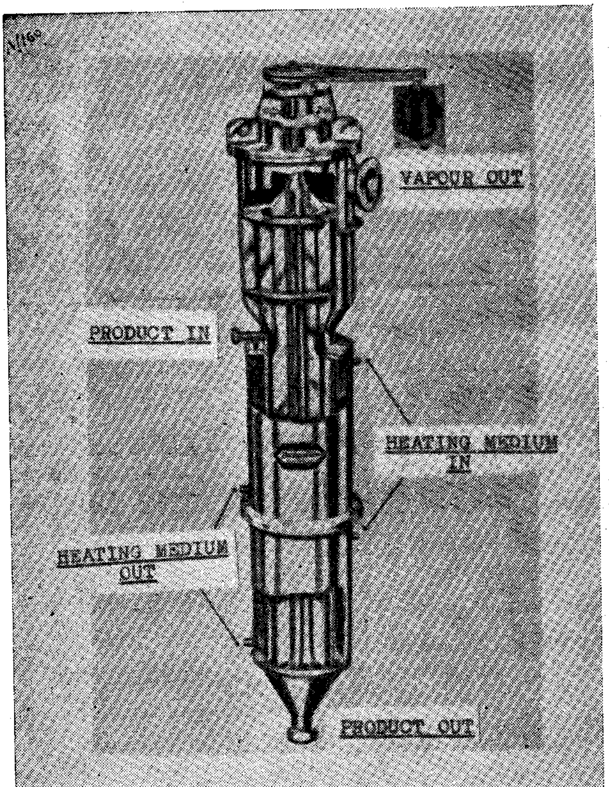

PLATE TURBULENT FILM SINGLE PASS EVAPORATOR

continuous. The concentration was completed in a single pass with very short heating period. The technical details of a typical run on this evaporator are presented in Table I.

Methods of Analysis

Sampling—About one gallon samples of orange concentrates of different degrees of concentration were drawn and analysed as follows—°*Brix* was determined by using a pocket refractometer at room temperature and later, the temperature correction and correction for citric acid[4] were applied. *Acidity* was determined by titrating known weights of concentrate against standard alkali, using phenolphthalin as an indicator and the results expressed as percent anhydrous citric acid W/W. *Brix/acid ratio* was obtained by dividing the corrected °Brix by % acidity. *pH* was determined by using glass-electrode Cambridge pH meter. *Concentration ratios* (V/V and W/W) of the concentrates in relation to single-strength juice were calculated as usual from °Brix and corresponding specific gravity of the juice and concentrates. *Viscosity* measurements were made with a Brook-Field Synchrolectric Viscometer[5] at constant temperature (20°C). *Colour* of concentrates was directly evaluated as per cent *spectral reflectance* with the help of a Hardy's Recording spectro-photometer fitted with the reflectance attachment.

TABLE 1

Technical Working Details of the Forced Circulation, Falling Film Evaporator

Serial No.	Particulars	Details
1	Steam Pressure	5·0 lb
2	Steam Temperature	235°F
3	Chamber Vacuum Hg.	750 mm.
4	Jacket Temperature	100—102°C
5	Vapour Temperature	24—30°C
6	Rotor Peripheral Speed ft/sec.	45
7	Feed rate lbs/hr	700
8	Vapour rate lbs/hr	500
9	Product Temperature (In)	32°C
10	Product Temperature (Out)	21°C
11	Cooling Water Temperature	
	(*a*) In	17·5°C
	(*b*) Out	74·0°C
12	Cooling Water Pressure	27½ lb.
13	Feed Composition (Orange Juice)	12° Brix
14	Orange Concentrate Composition	50° Brix
15	Orange Concentrate Yield lbs/hr	200
16	Time of Start .. }	Continuous operation
17	Time of Finish .. }	

Ascorbic acid was determined colorimetrically by the formaldehyde condensation method[6] and *Carotene* by the method of Association of Vitamin Chemists[7].

Results

The physico-chemical changes in orange concentrate at different stages during vacuum concentration are presented in Table 2. The pronounced changes in viscosity, ascorbic acid, and acidity are illustrated in Figure 1. The spectral reflectance curves (covering the wavelength range of 400-700 mμ), depicting the colour changes in concentrates, are presented in Figure 2.

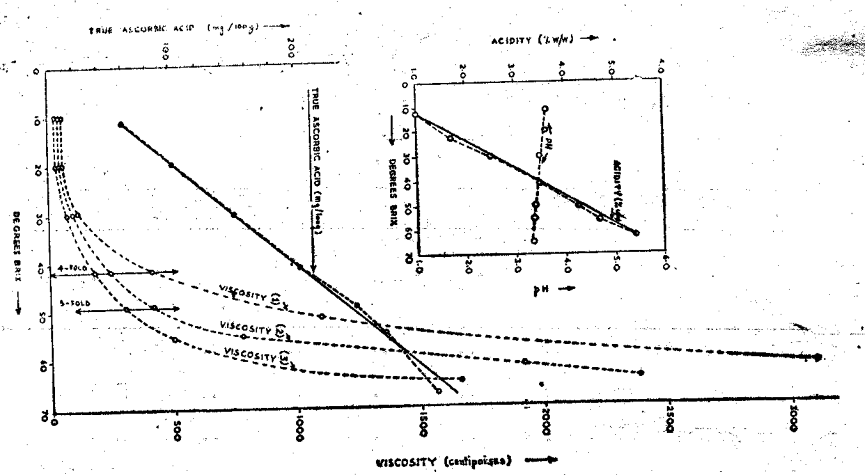

FIG. 1. VALENCIA ORANGE CONCENTRATE

Changes in Viscosity, Ascorbic, Acid, p^H and acidity in Orange Concentrate in relation to degree of concentration.

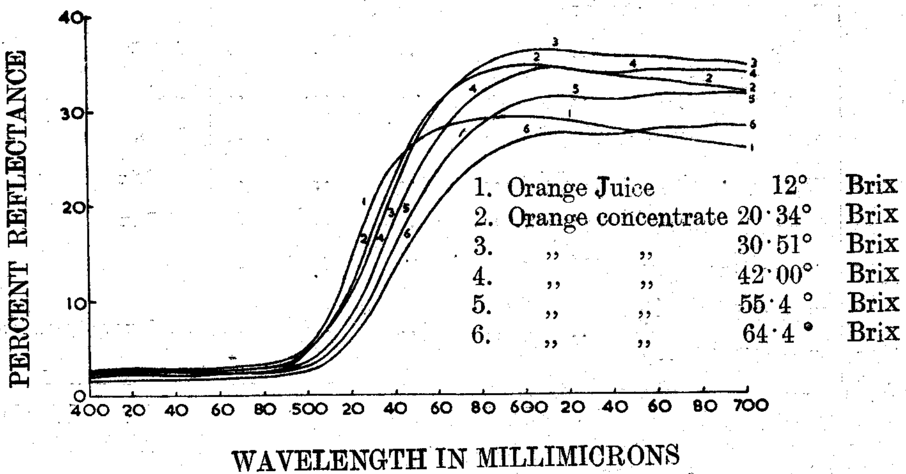

FIG. 2. SPECTRAL REFLECTANCE CURVES FOR VALENCIA ORANGE JUICE AT DIFFERENT STAGES OF CONCENTRATION.

(Measurements made with General Electric Recording Hardy's spectrophotometer equipped with reflectance attachment)

Discussion

A study of table 2 reveals that with the advancing concentration, there was a gradual increase in °Brix, acidity, viscosity, colour, ascorbic acid, and carotene, there being no change in °Brix/acid ratio but gradual, though slight, fall in *pH*.

TABLE 2

Physico-Chemical Changes in Valencia Orange Concentrate During Commercial Scale Vacuum Concentration

Serial No.	Physico-chemical Characteristics	Stages in Concentration						
		I	II	III	IV	V	VI	VII
1	Refractometric Solids %.	11·8	20·0	30·0	41·4	48·5	54·5	63·0
2	Acidity % W/W (As anhyd. citric acid).	1·00	1·71	2·57	3·54	4·15	4·66	5·39
3	Degree Brix (Corrected for acid).	12·00	20·34	30·51	42·09	49·30	55·40	64·04
4	Specific Gravity ..	1·047	1·083	1·129	1·187	1·227	1·260	1·311
5	Brix/acid ratio ..	12·00	12·00	12·00	12·00	12·00	12·00	12·00
6	pH	3·56	3·53	3·50	3·47	3·45	3·43	3·40
7	Concentration Ratio							
	(*a*) W/W ..	1·00	1·70	2·54	3·59	4·11	4·62	5·34
	(*b*) V/V ..	1·00	1·76	2·74	4·07	4·82	5·56	6·69
8	Viscosiy (Centipoises)	17·97	28·45	86·80	240	425	770	2380
9	Percent. Reflectance							
	(*a*) at 600 mμ ..	29·3	34·8	36·3	34·0	32·5	31·0	27·2
	(*b*) at 700 mμ ..	26·3	32·0	35·0	34·4	33·1	32·0	28·6
10*a*	True Ascorbic Acid mg/100g.	60·22	100·1	149·6	204·6	246·25	271·13	311·5
10*b*	True Ascorbic Acid as % of total.	99·50	98·5	97·9	95·5	95·89	96·35	94·91
10*c*	% Losses in Asc. Acid during concentration.	0·00	1·90	2·29	3·11	4·85	3·91	3·12

Viscosity—Up to 4-fold concentrate (stage IV, Table 2), the increase in viscosity was comparatively slight in all the three curves (1—3) for viscosity (representing early, middle and late season concentrates respectively) but after 5 fold concentration (stages VI and VII), there was a steep rise in viscosity (figure 1). Thus, while the viscosity of single-strength juice was 17·97 centipoises and that of 4 fold 240 centipoises, the corresponding values for stages V, VI and VII were 425, 770 and 2380 centipoises respectively. Further, it was also interesting to note that with the advancing season, there was a fall in the viscosity, probably because of the natural decrease in pectin content of the juice. In the light of the above observation, it would therefore be desirable to use mid-season oranges for concentrate manufacture. Further, these data also prove that it would not be advantageous to carry on concentration beyond 5-fold because of much higher viscosity with the consequent low rate of heat transfer and hence the inferior quality of the concentrate.

pH—The changes in *pH* were very slight—the figures for the single-strength juice, 4-fold and 6·7 fold concentrates being 3·56, 3·47 and 3·40 respectively. The corresponding values for percent acidity were 1·00, 3·54 and 5·39 respectively.

Ascorbic Acid—There was negligible amount of apparent ascorbic acid present in the fresh single-strength juice, while at different stages of concentration, it ranged from 1·5 to 5·09 per cent. The percent losses of ascorbic acid ranged from 1·90 to 4·85, the losses being not always consistent with the stages of concentration. At other occasions, negligible losses were noticed, probably because of extremely short period of concentration, the apparent variations being perhaps due to slight variations in the jacket temperature and in the time of exposure. In general, the losses were well within 5 per cent irrespective of the stages of concentration upto 63° Brix. These figures are much less than those reported recently by Siddappa and Bhatia[2] as 10·5 per cent up to 72° Brix concentration. The higher losses are obviously due partly to the high temperatures of concentrations employed by them (50—52°C) and partly due to varietal differences.

Carotene—There were negligible losses in Carotene during vacuum concentration of orange juice.

Colour—The dominant wavelength for single-strength juice, 4-fold and 6·7-fold concentrate, as worked out by the Hardy method[8], were found to be 578, 58?, 583 m*u* respectively. Obviously, there was a shift in the dominant wavelength during concentration of the juice. The *x*, *y* and *Y* values for these curves were as follows—

Colour Coordinates*	Orange Juice	Orange concentrate	
	118° Brix	42° Brix	62° Brix
x	0·465	0·497	0·505
y	0·457	0·445	0·441
Y	22·080	22·400	17·130

*Where x and y are the trichromatic coefficients and *Y* is the luminous reflectance (%). The C.I.E. values for Illuminant *C* were worked out by the selected ordinate method from the spectral reflectance curves 1, 4 and 6.

From the curves 1—6 (fig. 2), it would be interesting to note that there are 3 distinct stages in each curve. In the first stage, *i.e.*, between 400 and nearly 500 mμ, irrespective of the degree of concentration, there is negligible change in per cent reflectance. This part of the curve represents the blue component of the colour. In the second stage (500—600 mμ), all the curves are comparatively steep while in the third phase, *i.e.*, between 600—700 m μ, the curves were comparatively flat.

Further, between the wavelength range of 520—540 mμ, there was a gradual decrease in per cent reflectance corresponding to the increase in the degree of concentration of the juice. (Curves 1—5).

Up to 30° Brix concentration (Curves 1—3, there was a corresponding increase in per cent reflectance particularly at all wave-lengths in the third phase (600—700 mμ), but in concentrates from 42° to 64° Brix (Curves 4—6), there was a corresponding decrease in per cent reflectance. This indicates a deviation from the normal colour of the fresh juice, *i.e.*, slight darkening as a result of mild heat treatment during vacuum concentration.

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